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IS 11803 (1986): Magnesium oxide for explosive ammunition protective compositions and pyrotechnic industry [CHD 26: Explosives and Pyrotechnics]



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Indian Standard

SPECIFICATION FOR
MAGNESIUM OXIDE FOR EXPLOSIVE
AMMUNITION PROTECTIVE COMPOSITIONS
AND PYROTECHNIC INDUSTRY

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR MAGNESIUM OXIDE FOR EXPLOSIVE AMMUNITION PROTECTIVE COMPOSITIONS AND PYROTECHNIC INDUSTRY

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Indian Standard

SPECIFICATION FOR MAGNESIUM OXIDE FOR EXPLOSIVE AMMUNITION PROTECTIVE COMPOSITIONS AND PYROTECHNIC INDUSTRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 July 1986, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

0.2 Magnesium oxide is used as an ingredient in the manufacture of ammunition protective composition and in double base propellants. It is also used in the manufacture of lead styphnate and in explosive and pyrotechnic industry.

0.3 Indian Standards are already available on magnesium oxide (IS : 2529-1983* and IS : 9407-1980†) which cover the requirement of magnesium oxide for use in cosmetic and rubber industry.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960‡. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for magnesium oxide intended for use in explosive ammunition protective compositions and pyrotechnic industry.

*Specification for magnesium oxide for cosmetic industry (*second revision*).

†Specification for light magnesium oxide for rubber industry.

‡Rules for rounding off numerical values (*revised*).

2. GRADES

2.1 There shall be three grades of the material as follows:

- a) Grade I — for use in the manufacture of lead styphnate and propellants.
- b) Grade II — for use as an ingredient in the manufacture of ammunition protective composition (cements) and in double base propellants.
- c) Grade III — for use in explosive and pyrotechnic industry.

3. REQUIREMENTS

3.1 **Description** — The material shall be in the form of white powder, free from lumps and other visible impurities.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in column 6 of the table.

TABLE 1 REQUIREMENTS FOR MAGNESIUM OXIDE FOR EXPLOSIVE AMMUNITION PROTECTIVE COMPOSITIONS AND PYROTECHNIC INDUSTRY

SL No.	CHARACTERISTIC	REQUIREMENTS FOR GRADE			METHOD OF TEST, REF TO CL NO. IN APPENDIX A
		I	II	III	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Loss on ignition, percent by mass, <i>Max</i>	4.0	5.0	4.0	A-2
ii)	Matter insoluble in dilute hydrochloric acid, percent by mass, <i>Max</i>	0.1	0.2	4.5	A-3
iii)	Fineness, material retained on				A-4
	53 micron IS Sieve, percent by mass, <i>Max</i>	3.0	1.5	—	
	63 micron IS Sieve, percent by mass, <i>Max</i>	—	—	2.0	

(Continued)

**TABLE 1 REQUIREMENTS FOR MAGNESIUM OXIDE FOR EXPLOSIVE
AMMUNITION PROTECTIVE COMPOSITIONS AND PYROTECHNIC
INDUSTRY — *Contd***

SL No.	CHARACTERISTIC	REQUIREMENTS FOR GRADE			METHOD OF TEST, REF TO CL No. IN APPENDIX A
		I	II	III	
(1)	(2)	(3)	(4)	(5)	(6)
	125 micron IS Sieve, percent by mass, <i>Max</i>	Nil	—	—	
	250 micron IS Sieve, percent by mass, <i>Max</i>	—	Nil	—	
iv)	Calcium compound (as CaO), percent by mass, <i>Max</i>	1.0	1.0	2.0	A-5
v)	Carbonates (as $MgCO_3$), percent by mass, <i>Max</i>	—	—	2.5	A-6
vi)	Water soluble sulphates (as H_2SO_4), percent by mass, <i>Max</i>	0.20	0.20	0.5	A-7
vii)	Bulk density, g/ml, <i>Max</i>	—	—	0.6	A-8
viii)	Magnesium (as MgO), percent by mass, <i>Min</i>	95.0	92.0	89.0	A-9
ix)	Grit, insoluble in aqua regia and retained on 125 micron IS Sieve, percent by mass, <i>Max</i>	0.05	—	0.05	A-10
x)	Sodium compounds (as Na_2O), percent by mass, <i>Max</i>	0.25	—	—	A-11
xi)	Iron compound (as Fe), percent by mass, <i>Max</i>	0.01	—	—	A-12
xii)	Lead compounds (as $PbSO_4$)	Not to be traceable	—	—	A-13
xiii)	SiO_2 content and matter precipitated by ammonia, percent by mass, <i>Max</i>	—	0.1	—	A-14

(Continued)

TABLE 1 REQUIREMENTS FOR MAGNESIUM OXIDE FOR EXPLOSIVE AMMUNITION PROTECTIVE COMPOSITIONS AND PYROTECHNIC INDUSTRY — Contd

SL No.	CHARACTERISTIC	REQUIREMENTS FOR GRADE			METHOD OF TEST, REF TO CL No. IN APPENDIX A
		I	II	III	
(1)	(2)	(3)	(4)	(5)	(6)
xiv)	R ₂ O ₃ content, percent by mass, <i>Max</i>	—	0.3	—	A-15
xv)	Ammonium compound (as NH ₃)	Not to be traceable	—	—	A-16
xvi)	Matter soluble in water, percent by mass, <i>Max</i>	—	0.5	—	A-17
xvii)	Water soluble chlorides (as Cl), percent by mass, <i>Max</i>	0.03	0.4	0.5	A-18
xviii)	Moisture, percent by mass, <i>Max</i>	1.0	—	—	A-19

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in polyethylene lined bags or in such other containers as agreed to between the purchaser and the supplier.

4.2 Marking — The containers shall be securely closed and legibly and indelibly marked with the following information:

- a) Name and grade of the material;
- b) Mass of the material in the container;
- c) Manufacturer's name and/or recognized trade-mark, if any; and
- d) Lot number to enable the batch of manufacture to be traced from the records.

4.2.1 The containers may also be marked with the Standard Mark.

NOTE—The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 Rules and Regulations made thereunder. The BIS Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. BIS marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the BIS Certification Mark may be granted to manufacturers or processors, may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 The method of drawing representative sample from a lot and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Appendix B.

APPENDIX A

(Clause 3.2 and Table 1)

METHODS OF TEST FOR MAGNESIUM OXIDE FOR AMMUNITION PROTECTIVE COMPOSITIONS AND PYROTECHNIC AND EXPLOSIVE INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF LOSS ON IGNITION

A-2.1 Transfer about 2 g of the sample to a clean, dry and weighed (M_1) silica crucible and weigh accurately (M_2). Heat gently on a Bunsen flame for about 30 minutes and then to red heat (600 to 700°C) in a muffle furnace. Remove from the furnace, cool it outside till it is safe to be transferred to a desiccator. Cool to room temperature in a desiccator and reweigh to constant mass (M_3).

$$\text{Loss on ignition, percent by mass} = \frac{M_2 - M_3}{M_2 - M_1} \times 100$$

*Specification for water for general laboratory use (*second revision*).

A-3. DETERMINATION OF MATTER INSOLUBLE IN HYDROCHLORIC ACID

A-3.1 Reagents

A-3.1.1 *Dilute Hydrochloric Acid* — approximately 0.2 N.

A-3.2 Procedure — Weigh accurately about 2 g of the material. Transfer it to a round bottom flask with 100 ml of dilute hydrochloric acid. Fix up a reflux condenser and boil the material on a sand-bath for 5 minutes. Cool and filter through a sintered glass crucible No. G 4. Wash free from chlorides, dry the residue in an oven at 105 to 110°C to constant mass, cool and weigh.

A-3.3 Calculation

$$\text{Matter insoluble in hydrochloric acid, percent by mass} = 100 \times \frac{M}{M_1}$$

where

M = mass in g of the residue, and

M_1 = mass in g of the material taken for the test.

A-4. DETERMINATION OF FINENESS

A-4.1 Procedure — Place 10 g of the material on an IS sieve of required size and brush it gently with a 25-mm varnish brush for 15 minutes or until no further material passes through the sieve, whichever is the lesser period. Remove the sieve and weigh the portion of the sample retained on it.

A-4.2 The material shall be considered to have passed the requirement of the test if there is no residue left on the sieve.

A-5. DETERMINATION OF CALCIUM COMPOUND

A-5.0 Two methods have been prescribed, namely, gravimetric method and volumetric method. In case of dispute, the volumetric method shall be the referee method.

A-5.1 Gravimetric Method

A-5.1.1 Reagents

A-5.1.1.1 *Concentrated hydrochloric acid* — see IS : 265-1976*.

*Specification for hydrochloric acid (second revision).

A-5.1.1.2 Rectified spirit — 95 percent by volume.

A-5.1.1.3 Sulphuric acid — 25 percent (*m/v*).

A-5.1.2 Procedure — Dissolve about 0.5 g of freshly ignited material, accurately weighed, in concentrated hydrochloric acid added in small portions. Filter, if necessary, add to the filtrate 100 ml of rectified spirit and 40 ml of sulphuric acid and let it stand overnight. If crystals of magnesium sulphate separate out, warm the mixture to about 50°C to dissolve them. Filter, wash with a mixture of two volumes of rectified spirit and one volume of sulphuric acid and ignite. Weigh the residue.

A-5.1.3 Calculation

Calcium (as CaO), percent by mass of the ignited residue = $\frac{41.18 A}{M}$

where

A = mass in g of the residue, and

M = mass in g of the ignited material taken for the test.

A-5.2 EDTA Method

A-5.2.1 Outline of the Method — Calcium, like some other metal ions, is complexed by EDTA by choosing the proper pH and indicator. Using calcon as indicator, calcium can be estimated in the presence of magnesium since magnesium is precipitated quantitatively as magnesium hydroxide in the pH range of the reaction.

A-5.2.2 Reagents

A-5.2.2.1 Diethylamine

A-5.2.2.2 Calcon indicator — prepared by dissolving 0.2 g of the dye-stuff in 50 ml of methyl alcohol.

A-5.2.2.3 Standard EDTA (disodium dihydrogen ethylenediamine tetra acetate solution) — Weigh 3.772 5 g of EDTA, dissolve in water free from polyvalent ions (distilled water for this purpose should be passed through a column of cation exchange resins in the sodium form) and make up to a volume of 1 000 ml. Concentration of the resulting solution will be 0.01 M. (If the EDTA is not of analytical reagent grade, then the solution prepared needs to be standardized against zinc chloride solution or magnesium chloride solution which is prepared from analytical reagent grade material or corresponding metal pellets of analytical reagent grade.)

A-5.2.2.4 Sample solution — Weigh about one gram of the material accurately, dissolve in water free from polyvalent ions, filter and make up to 100 ml.

A-5.2.3 Procedure — Pipette 10 ml of the sample solution (*see* A-5.2.2.4) into 250-ml conical flask. Add about 40 ml of water and 5 ml of diethylamine, giving the mixture a pH of about 12.5. Under these conditions, magnesium, if present, precipitates quantitatively as the hydroxide. Add four drops of calcon indicator to the solution and titrate with standard EDTA solution with shaking until the colour changes from pink to a pure blue.

A-5.2.4 Calculation

$$\text{Calcium (as CaO), percent by mass} = \frac{0.56 \times VM}{A}$$

where

V = volume in ml of standard EDTA solution,

M = molarity of standard EDTA solution, and

A = mass in g of material taken (A-5.2.2.4).

A-6. DETERMINATION OF CARBONATES

A-6.0 Outline of the Method — The material is heated with sufficient water and the carbonates in aqueous suspension are reacted with a known excess of standard hydrochloric acid. The excess acid is then back titrated with standard sodium hydroxide solution and the carbonate is calculated as magnesium carbonate.

A-6.1 Reagents

A-6.1.1 Standard Hydrochloric Acid — 0.1 N.

A-6.1.2 Standard Sodium Hydroxide Solution — 0.1 N.

A-6.1.3 Bromophenol Blue Indicator Solution — 0.1 g in 100 ml of rectified spirit.

A-6.2 Procedure — Weigh accurately about 10 g of the material in a 500-ml conical flask and add about 200 ml of previously boiled water. Run in from a burette, known excess of standard hydrochloric acid. Titrate the excess acid with standard sodium hydroxide solution using bromophenol blue as indicator. Carry out side by side a blank determination exactly in the same way.

A-6.3 Calculation

$$\text{Carbonate (as MgCO}_3 \text{), percent by mass} = \frac{(A - B) \times N \times 42 \times 100}{1\,000 \times M}$$

where

A = volume in ml of standard sodium hydroxide solution required for blank,

B = volume in ml of standard sodium hydroxide solution required for sample,

N = normality of standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

A-7. DETERMINATION OF SULPHATES

A-7.1 Reagents

A-7.1.1 *Concentrated Hydrochloric Acid* — see IS : 265-1976*.

A-7.1.2 *Dilute Hydrochloric Acid* — approximately 10 percent (m/v).

A-7.1.3 *Barium Chloride Solution* — approximately 10 percent (m/v).

A-7.2 Preparation of Test Samples

A-7.2.1 From each of the containers selected, draw a small representative portion of the material not less than 200 g in mass.

A-7.2.2 Out of these portions, an equal quantity of the material shall be taken and mixed thoroughly to form a composite test sample of mass not less than 0.5 kg. The composite test sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

A-7.2.3 The remaining portion of the material from each container shall be divided into three equal parts and transferred to separate bottles, giving full identification particulars of the samples on the bottles. The material in each bottle constitutes an individual test sample. One of these three sets (each set containing one bottle representing each container sampled) shall be marked for the purchaser, another for the supplier and the third for the referee.

A-7.2.4 The referee test samples consisting of a composite test sample and a set of individual test samples shall bear the seal of both the purchaser

*Specification for hydrochloric acid (*second revision*).

and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier and shall be used in the case of a dispute between the two.

A-7.3 Procedure — Weigh accurately about 5 g of the *prepared sample* and dissolve in 50 ml of water. Add 15 ml of concentrated hydrochloric acid and evaporate the solution to dryness. Moisten the residue with 10 ml of dilute hydrochloric acid, dilute to 200 ml with water and filter, if necessary. Heat the solution to boiling and add drop by drop, 10 ml of barium chloride solution. Boil the solution for 15 minutes and then allow to stand for four hours. Filter through a tared Gooch crucible. Wash the precipitate thoroughly with hot water till free from chlorides and dry to constant weight at 105 to 110°C.

A-7.4 Calculation

$$\text{Sulphates (as H}_2\text{SO}_4 \text{), percent by mass} = 49 \frac{M_1}{M}$$

where

M_1 = mass in g of the precipitate obtained, and

M = mass in g of the *prepared sample* taken for the test.

A-8. DETERMINATION OF BULK DENSITY

A-8.1 Apparatus

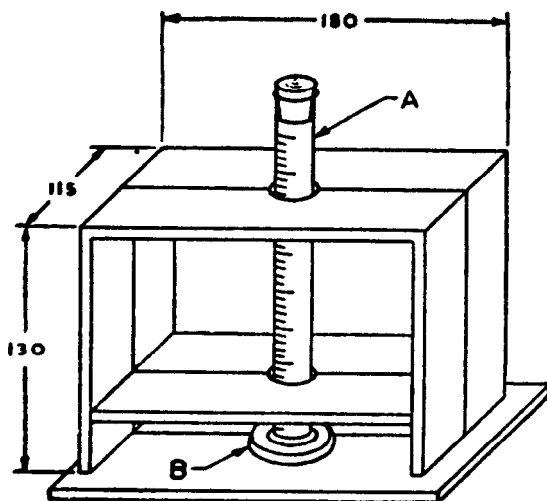
A-8.1.1 Assemble the apparatus as shown in Fig. 1. The measuring cylinder *A* shall be of 250 ml capacity and shall conform to IS : 878-1975*. The base of the measuring cylinder shall be ground flat. The distance between the flat-ground part of the base of the measuring cylinder *A* and the rubber base pad *B*, when the measuring cylinder *A* is raised to the full height, shall be 25 ± 2 mm.

A-8.1.2 The rubber base pad *B* shall have a shore hardness of 42 to 50.

A-8.1.3 Pans of the balance shall be at least 10 cm in diameter and the balance shall be sensitive to less than 0.1 g.

A-8.2 Procedure — Place 50 g of the material in the graduated cylinder fitted in the apparatus and tap it until a constant volume of the material is obtained. Note down the volume of the material.

*Specification for graduated measuring cylinders (*first revision*).



All dimensions in millimetres.

FIG. 1 APPARATUS FOR THE DETERMINATION OF BULK DENSITY

A-8.3 Calculation

$$\text{Bulk density, g/ml} = \frac{M}{V}$$

where

M = mass in g of the material taken for the test, and

V = volume of the material in the cylinder.

A-9. DETERMINATION OF MAGNESIUM

A-9.0 Two methods are prescribed. In case of dispute, EDTA method shall be the referee method.

A-9.1 Method I

A-9.1.1 Reagents

A-9.1.1.1 Standard sulphuric acid — 1 N.

A-9.1.1.2 Standard sodium hydroxide solution — 1 N, freshly standardized.

A-9.1.1.3 Methyl orange indicator solution — Dissolve 0.01 g of methyl orange in 100 ml of water.

A-9.1.2 Procedure — Weigh accurately about 1 g of the freshly ignited material and transfer it to a 250-ml conical flask. Add into the flask about 20 ml of water and transfer with a pipette 25 ml of standard sulphuric acid. Cover the flask with a watch-glass and stir the flask carefully. Wash down the watch-glass and sides of the conical flask with water and titrate the solution in the flask with standard sodium hydroxide solution using methyl orange indicator. Carry out a blank determination using the same quantities of the reagents.

A-9.1.3 Calculation

$$\text{Magnesium (as MgO), percent by mass of the ignited residue} = \frac{2.016 (V_1 - V_2) N}{M} - 0.719 A$$

where

V_1 = volume in ml of standard sodium hydroxide solution required for titration in the blank determination;

V_2 = volume in ml of standard sodium hydroxide solution, required for titration in the test with the material;

N = normality of standard sodium hydroxide solution;

M = mass in g of the ignited material taken for the test; and

A = percentage by mass of calcium (as CaO) as obtained in A-5.2 .

A-9.2 Method II (EDTA Method)

A-9.2.1 Outline of the Method — Calcium is precipitated as calcium oxalate and magnesium, if any, is estimated by complexing with EDTA using eriochrome black T.

A-9.2.2 Reagents

A-9.2.2.1 EDTA — 0.01 M (see A-5.2.2.3).

A-9.2.2.2 Eriochrome black T — Dissolve 0.2 g of the dyestuff in 15 ml of triethanolamine and 5 ml of absolute ethanol.

A-9.2.2.3 Standard magnesium solution — 0.01 M. Weigh 6.1 g of magnesium sulphate heptahydrate and dissolve in 50 ml of water. Make up to 250 ml with water in a volumetric flask.

A-9.2.2.4 Ammonium oxalate

A-9.2.2.5 Ammonium chloride

A-9.2.2.6 Dilute ammonium hydroxide — (1 : 1 by volume).

A-9.2.2.7 Ammonia — ammonium chloride buffer solution — pH 10. Add 142 ml of concentrated ammonium hydroxide to 17.5 g of ammonium chloride and dilute to 250 ml with water.

A-9.2.2.8 Dilute hydrochloric acid — 1 : 1 by volume.

A-9.2.2.9 Calcium precipitating buffer solution — Dissolve 6 g of ammonium oxalate in about 100 ml of water, add 144 g of ammonium chloride and 13 ml of concentrated ammonium hydroxide and dilute the resulting solution to 1 litre.

A-9.2.3 Standardization of EDTA Solution — Pipette 25 ml of standard magnesium solution into a conical flask and dilute to 100 ml with water. Add 5 ml of buffer followed by the addition of 6 drops of eriochrome black T indicator and titrate with EDTA solution until the colour changes from red to pure blue.

1 ml of 0.01 M EDTA = 0.243 2 mg of Mg.

A-9.2.3.1 Procedure — Weigh 1 g of magnesium oxide into a 250-ml beaker and add 40 ml of water. Add 5 ml of dilute hydrochloric acid slowly with constant stirring. Once the material is dissolved, add another 5 ml of dilute hydrochloric acid. Add a few drops of methyl red indicator. Heat the solution to boiling. Add a clear solution of 6 g of ammonium oxalate dissolved in 100 ml of water slowly with constant stirring. To the hot solution (70-80°C), add dilute ammonium hydroxide 1 : 1 dropwise until the solution becomes fairly alkaline. Digest on water bath for 1 hour.

A-9.2.3.2 Decant the supernatant liquid through No. 42 Whatman filter paper and collect the filtrate. Transfer the precipitate quantitatively to the filter paper and wash three times with 25-30 ml of water. Collect the washings along with the filtrate. Evaporate the solution to 100 ml. Add 2 ml of buffer solution followed by the addition of 3-4 drops of eriochrome black T indicator. Titrate the solution with EDTA solution at 40°C until the colour changes from red to pure blue. (The last traces of reddish shade should disappear at the end point. Complex formation does not take place instantaneously. Titration, therefore, should be conducted slowly near the end point.)

A-9.2.4 Calculation — Calculate magnesium as magnesium nitrate on the basis that one millilitre of 0.01 M EDTA solution is equivalent to 1.096 4 of magnesium nitrate [Mg (NO₃)₂].

A-10. DETERMINATION OF GRIT

A-10.1 Matter Insoluble in Dilute Hydrochloric Acid

A-10.1.1 Reagents

- a) *Dilute hydrochloric acid* — approximately 0.2 N.

A-10.1.2 Procedure — Brush the residue obtained in A-4 on 63-micron IS Sieve and weigh the material retained.

A-10.2 Matter Insoluble in Aqua Regia

A-10.2.1 Reagents

- a) *Aqua regia* — prepared by mixing 3 volumes of concentrated hydrochloric acid and one volume of concentrated nitric acid.

A-10.2.2 Procedure — Weigh accurately 10 g of the sample and dissolve by stirring in 30 ml of aqua regia. Heat to boiling and dilute with water to 100 ml. Filter through a tared filter paper, wash the residue thoroughly with water till free from chlorides and dry at 105 to 110°C to constant mass. Transfer the residue on the filter paper to a 125-micron IS Sieve with a light camel hair brush and weigh the material retained.

A-11. DETERMINATION OF SODIUM COMPOUNDS

A-11.1 Weigh about 3 g of the sample accurately, dissolve it in hydrochloric acid and dilute it to 150 ml with water. Treat the sample solution with 15 ml of zinc uranyl acetate reagent and stir vigorously, preferably mechanically for at least 30 minutes. Allow to stand for 1 hour and filter through a weighed sintered glass G4 crucible. Wash the precipitate four times with 2 ml portions of the precipitating reagent and then 10 times with 95 percent alcohol saturated with sodium zinc uranyl acetate at room temperature and finally with dry ether or acetone. Dry for 30 minutes at 105°C. Weigh as $\text{NaZn}(\text{UO}_2)_3(\text{C}_2\text{H}_3\text{O}_2)_9 \cdot 6\text{H}_2\text{O}$.

A-11.2 Alternatively, the sodium compounds may be determined by flame photometer.

A-12. DETERMINATION OF IRON

A-12.1 Weigh about 2 g of sample accurately, dissolve in hydrochloric acid, dilute it and reduce the same with stannous chloride solution. Add 200 ml of 2.5 percent sulphuric acid, 5 ml of syrupy phosphoric acid and 0.3-0.4 ml of sodium diphenylamine sulphonate or diphenylamine indicator. Titrate rapidly with constant stirring against standard 0.1 N potassium dichromate to the first permanent violet-blue colouration.

A-13. DETERMINATION OF LEAD

A-13.1 Weigh about 0.3 g of material accurately, dissolve it in about 20 ml of water, add 2 ml of pure concentrated sulphuric acid, evaporate the mixture as far as possible on the water bath, then on a sand-bath, wire-gauze or low-temperature hot-plate until thick white fumes of sulphuric acid are freely evolved. Cool, dilute carefully with 40 ml of water (the final solution should contain about 4 ml of concentrated sulphuric acid in 100 ml of solution), mix thoroughly, and allow to stand for at least an hour. Filter off the precipitate of lead sulphate through a weighed porcelain or silica gooch crucible. Wash the precipitate three times with 3 percent (by volume) sulphuric acid saturated with lead sulphate at room temperature. Finally wash with rectified spirit until free from sulphate, and then dry at 130°C to constant weight. Weigh as lead sulphate.

A-14. DETERMINATION OF SiO_2 CONTENT AND MATTER PRECIPITATED BY AMMONIA

A-14.1 Transfer about 5 g of the sample, accurately weighed, to a clean, dry platinum dish, add 50 ml of distilled water followed by gentle addition of 30 ml of dilute sulphuric acid (1:3). Evaporate the solution assisted by occasional stirring with a glass rod, first on a boiling water bath and then on a sand bath till all sulphuric acid fumes disappear (avoid evaporation to dryness). Dissolve the residue in 150 ml distilled water by heating and transfer to a clean, dry 500-ml beaker. Add 1 ml of 25 percent nitric acid and heat to boil for about 4-5 minutes. Add about 3 g ammonium chloride and 3-4 drops of methyl red indicator (0.1 percent solution in alcohol). Add 10 percent ammonia solution till it changes to yellow colour. Make up the volume of the solution to about 200 ml, heat for about 1 hour on a boiling water bath and filter through a blue filter paper (retain the filtrate for R_2O_3 estimation). Rinse the residue on filter paper till free from sulphates. Transfer the filter paper with residue to a previously ignited and tared platinum dish, dry in an oven at 105-110°C and further ignite in an electric muffle at 800-900°C. Cool outside to a temperature when it is safe to be transferred to a desiccator, further cool in a desiccator to room temperature and reweigh.

$$\text{Percentage of } \text{SiO}_2 \text{ content and matter precipitated by ammonia} = \frac{(\text{mass of residue after ignition})}{\text{mass of sample}} \times 100$$

A-15. DETERMINATION OF R_2O_3 CONTENT

A-15.1 Add into a platinum dish, after SiO_2 separation, a few drops of concentrated hydrochloric acid. Heat over a sand bath. Add this with filtrate after SiO_2 separation. Solution must be clear. Bring the solution

to 200 ml volume, heat the solution, add immediately a few drops of concentrated nitric acid (because of oxidation from ferrous to ferric form), 5 g ammonium chloride and 3-4 drops of methyl red indicator (the colour changes to red due to acidity of the solution). Add ammonium hydroxide till neutralized (changes to yellow). Boil, place over a boiling water bath to obtain the precipitate, and then filter through a black filter paper, rinse the precipitate with hot distilled water. Place the filter paper with the precipitate into a previously ignited, cooled, dry, tared platinum dish (M_1) and heat in an oven at $105-110^{\circ}\text{C}$ and further ignite in an electric muffle at $800-900^{\circ}\text{C}$. Cool outside to a temperature when it is safe to be transferred to a desiccator, further cool in a desiccator to room temperature and reweigh (M_2). (The content of the total oxides is calculated as percentage R_2O_3 content on the original sample.)

$$\text{Percentage } \text{R}_2\text{O}_3 \text{ content} = \frac{M_2 - M_1}{\text{mass of sample}} \times 100$$

A-16. DETERMINATION OF AMMONIA AND AMMONIUM COMPOUNDS

A-16.1 Reagents

A-16.1.1 Nessler Solution

A-16.1.2 Ammonium Chloride Solution (0.15 g per litre).

A-16.2 Procedure — 5 g of the sample is dissolved in water and the volume of the solution is made up to 100 ml in a Nessler tube. Two ml of Nessler's solution is added and any colour which develops is compared at the end of 3 minutes with that in other tubes containing known volumes of the standard ammonium chloride solution treated in a similar manner.

1 ml NH_4Cl solution = 0.001 percent NH_3 (on 5 g sample) (0.15 g per litre)

A-17. DETERMINATION OF MATTER SOLUBLE IN WATER

A-17.1 Procedure — Weigh accurately about 5 g of the material and place it in a 500-ml beaker. Add 100 ml of water and stir thoroughly. Cover the beaker with a watch-glass and allow to stand for 4 hours at room temperature, with occasional stirring. Filter into a 250-ml volumetric flask, wash the sample and filter paper with water and make up the volume to 250 ml. Transfer 125 ml of this solution to a tared evaporating dish and evaporate to dryness over a water-bath. Dry the residue at $105 \pm 2^{\circ}\text{C}$ and weigh till a constant weight is obtained.

A-17.2 Calculation

$$\text{Matter soluble in water, percent by mass} = \frac{200 M_2}{M_1}$$

where

M_2 = mass in g of the residue, and

M_1 = mass in g of the material taken for test.

A-18. DETERMINATION OF WATER SOLUBLE CHLORIDES

A-18.1 Reagents

A-18.1.1 *Dilute Nitric Acid* — 1 : 4 (v/v).

A-18.1.2 *Standard Silver Nitrate Solution* — 0.05 N.

A-18.1.3 *Potassium Chromate* — saturated solution.

A-18.2 Procedure — Weigh accurately about 5 g of the sample in a conical flask. Add to the flask 100 ml of water and shake the solution. Filter through a No. 40 Whatman filter paper. Wash the residue twice using about 5 to 10 ml of water. Transfer the solution to a porcelain dish and make it neutral to phenolphthalein either with sodium carbonate (Na_2CO_3) or with dilute nitric acid. Add two drops of saturated solution of potassium chromate (free from chloride). Titrate with 0.05 N standard solution of silver nitrate until a faint but permanent reddish tinge appears showing that all the chloride has combined with silver and the slight excess has formed a precipitate of silver chromate (Ag_2CrO_4). Carry out blank using all the reagents and apply the necessary correction.

A-18.3 Calculation

1 ml of 1 N AgNO_3 = 0.035 46 g NaCl

$$\text{Chlorides, percent by mass} = \frac{V \times N \times F \times 0.035\ 46 \times 100}{M}$$

where

V = volume in ml of standard silver nitrate solution required in titration,

N = normality of the standard silver nitrate solution used,

F = factor of standard silver nitrate solution, and

M = mass of the sample taken.

NOTE — The reaction is delicate, the detection of the end point is facilitated by frequent comparisons of the end contents of the porcelain dish in which the determination is being made with that of another dish placed alongside containing the same quantity of potassium chromate solution and the same quantity of water equal to the solution which has been titrated to a suitable end point.

A-19. DETERMINATION OF MOISTURE

A-19.1 Procedure — Weigh accurately about 1.0 g of the sample into a weighing bottle which has been dried, cooled in the desiccator and then weighed. Place the weighing bottle in the air oven for about 1 hour at $105 \pm 5^\circ\text{C}$. Remove the weighing bottle from the oven, cool in a desiccator to room temperature and weigh. Repeat this procedure. Keep the weighing bottle in the oven only for half an hour each time until the difference between two successive weighings is less than 1mg.

A-19.2 Calculation

$$\text{Moisture content, percent by mass} = \frac{100 \times M_1}{M}$$

where

M_1 = loss in mass in g of the sample upon drying, and

M = mass in g of the sample taken for the test.

APPENDIX B

(Clause 5.1)

SAMPLING OF MAGNESIUM OXIDE**B-1. GENERAL REQUIREMENTS OF SAMPLING**

B-1.1 For general requirements of sampling, the methods given in IS : 8883 (Part 1) - 1978* may be followed.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material of the same grade drawn from a single batch of processing shall constitute a lot. If a consignment is declared or known to consist of different batches of processing, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

B-2.1.1 Tests for determining the conformity of the lot to the requirements of the specification shall be done on each lot separately.

*Methods of sampling chemicals and chemical products : Part 1 General requirements and precautions.

B-2.2 The number of containers to be selected shall depend upon the size of the lot and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED

LOT SIZE	NUMBER OF CONTAINERS TO BE SELECTED
(1)	(2)
Up to 50	3
51 to 150	4
151 to 300	5
301 to 500	7
501 and above	10

B-2.3 The containers shall be selected from the lot at random and in order to ensure randomness of selection, the method given in IS : 4905-1968* may be followed.

B-3. NUMBER OF TESTS

B-3.1 Tests for all characteristics shall be conducted on composite sample.

B-4. CRITERIA FOR CONFORMITY

B-4.1 The lot shall be declared as conforming to the requirements of the specification if the composite sample fails in none of the tests conducted.

*Methods for random sampling.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²